TOOELE CHEMICAL AGENT DISPOSAL FACILITY (TOCDF)

DEMONSTRATION TEST PLAN FOR THE

AUTOCLAVE SYSTEM

APPENDIX C

ADT QUALITY ASSURANCE PROJECT PLAN

Revision 0

October 21, 2008

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Section 1.0

Autoclave Evaluation Test Report,
Continental Research and Engineering. LLC,
Englewood, CO, Revision 0,
April 21, 2008.

CONTINENTAL RESEARCH AND ENGINEERING, LLC

Autoclave Evaluation Test report

07017

Revision 0

April 21, 2008

Prepared for
EG&G Defense Materials, Inc.

By

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I. Executive Summary

An autoclave test was developed and executed[1] to determine the effectiveness of an autoclave to neutralize an agent surrogate applied to various secondary waste materials. This report documents and presents the results of the completed Autoclave Evaluation Test Plan. The test results demonstrate the ability of an autoclave to decontaminate secondary wastes from chemical warfare weapons demilitarization facilities.

The testing was conducted to evaluate autoclave technology as an alternate technology for decontamination of secondary wastes at TOCDF. The tests were conducted utilizing five waste materials: Charcoal, DPE suits, Soil, wood and Tap Gear. The wastes were contaminated with the agent surrogate material chloromethylnaphthalene. The surrogate contaminated wastes were treated in an autoclave at various temperatures to determine destruction removal efficiency of the surrogate material. Minicams measurement of surrogate levels was conducted on the autoclave headspace after each test. Extraction analysis was conducted on contaminated samples (sachets) spiked at both a high and low levels contained in each of the processed loads. Extraction analysis of all samples (sachets) spiked at a low level did not detect any surrogate material remaining. Because of this, only the samples (sachets) spiked at a high level were used in calculating DREs.

Decontamination tests on charcoal in both bulk and contained in a HVAC filter tray were successful. Minicams readings on the head space were very low and extraction analysis indicated a DRE of 99.99 to 99.999 %. Tests were conducted at 190°F for a period of 1 hour.

Decontamination tests on soil were successful. Minicams readings on the head space indicated that the surrogate was present in the vapor state at completion of the test. Extraction analysis indicated a DRE of 99.999% or better. Tests were conducted at 190°F for a period of 1 hour.

Decontamination tests on wood were successful. Minicams readings on the head space indicated that the surrogate was present in the vapor state at completion of the test. Extraction analysis indicated a DRE of 99. 9% on one sachet and the rest indicating 99.99% or better. The wood test was conducted with a full bin approximately 120 lbs. The test was conducted at 250°F for a period of 2 hrs and 30 minutes. There is the potential to increase the DRE by increasing processing temperature.

Decontamination tests on DPE suits and TAP gear were successful. Extraction analysis indicated a DRE in the range of 99% or above in most cases, with one test run at below the 99% DRE. The DPE suit tests were run at varying temperatures and times in an attempt to increase the DRE. The test temperature for DPE suits was increased to 280°F without degradation of the suits. It may be possible to treat the DPE suits at 290°F or higher without melting the suits.

It became apparent during the testing that the surrogate was not fully hydrolyzing before vaporizing and was not being hydrolized in the gas phase. This may be due to the surrogate selection or the selection of the carrier fluid (toluene). The testing demonstrates that secondary waste can be decontaminated in an autoclave. It is recommended that additional laboratory testing be completed using chemical agents and chloromethylnaphthalene without a carrier fluid. These tests should be used to further clarify the results of the surrogate tests and to identify the differences in reactions between the surrogate and chemical agents.

[1] Autoclave Evaluation Test Plan, Revision 3, CRE Project No. 07017 dated February 29, 2008

II. Introduction

The Tooele Chemical Agent Disposal Facility (TOCDF) is currently planning for processing of secondary and closure wastes. The site has sufficient time to implement changes that will avoid schedule impacts created by processing secondary wastes. The additional time allows the review and consideration of alternate technologies and addition of equipment to improve secondary waste processing. Installation of a system specifically for processing of secondary and closure wastes would allow parallel processing of secondary wastes, increase the availability of the Metal Parts Furnace (MPF) to process munitions, and shorten the facility's overall schedule. There is significant risk to the facility's schedule if alternate techniques (i.e., utilizing processing systems dedicated to secondary wastes) are not utilized for processing secondary waste.

Continental Research & Engineering (CR&E) completed an investigation of processing alternatives to reduce the schedule impact resulting from processing secondary waste through the existing demilitarization furnaces. Treatment of waste through an autoclave is an attractive processing alternative due to its relative low cost and short implementation schedule. Autoclaves have been successfully used for decontamination of BDO suits contaminated with chemical agents GB, VX and HD[2].

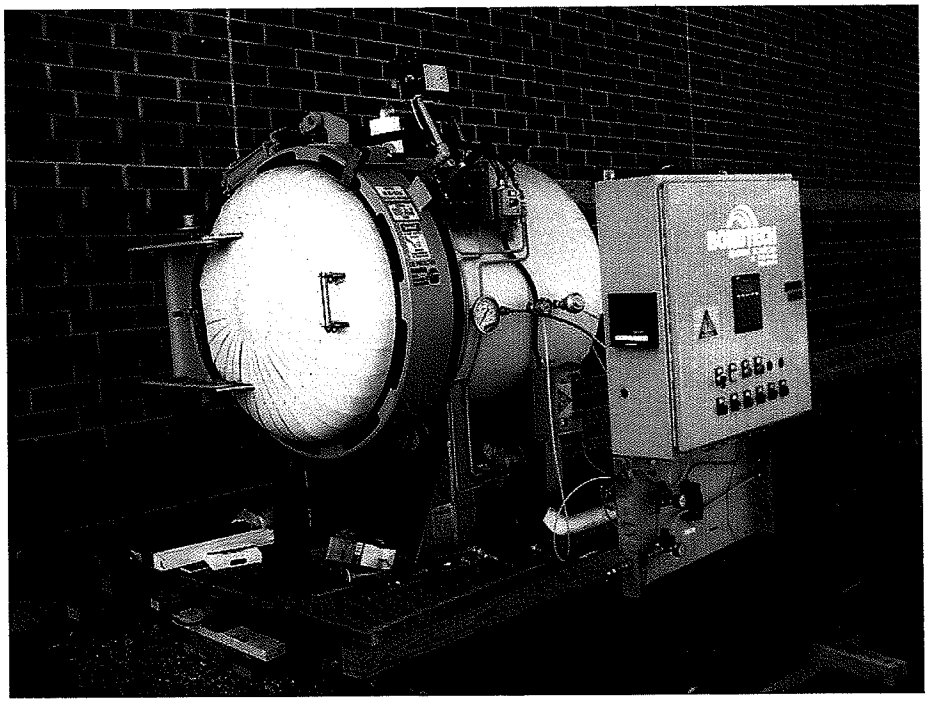
An autoclave test was developed and executed[3] to determine the effectiveness of an autoclave to neutralize an agent surrogate applied to various secondary waste materials. This report documents and presents the results of the completed Autoclave Evaluation Test Plan. The test results demonstrate the ability of an autoclave to decontaminate chemical warfare weapons demilitarization facilities secondary wastes.

III. Process Description

Autoclaves are designed to decontaminate materials by subjecting them to steam at high temperature and pressure. The contact of steam at high temperature and pressure with an agent surrogate is intended to neutralize the surrogate through hydrolysis in the same manner that chemical agents would decompose. All waste is fed to the autoclave through an autoclave inlet door. Waste is either bulk loaded into waste processing carts or loaded into processing trays and placed within the autoclave. Size of the autoclave and cart design determines the loaded autoclave capacity. The test autoclave had a capacity of one cart approximately 24"x 36"x 18" deep or three trays approximately 24" x 24" x 2" deep. A drawing and specifications for the autoclave and cart / tray system are contained in the Test Plan.

[2] Battle Dress Overgarment (BDO) Decontamination Test, U.S. Army Dugway Proving Ground, Dugway, UT Dec. 1992 and Battledress Overgarment (BDO) Decontamination Test - Follow-On Testing, U.S. Army Dugway Proving Ground, Dugway, UT July 1993.

[3] Autoclave Evaluation Test Plan, Revision 3, CRE Project No. 07017 dated February 29, 2008



Test Autoclave

The decontamination process is started following loading of the autoclave and securing of the inlet door by creating a vacuum within the autoclave vessel. The vacuum removes residual air from the autoclave vessel and aids in the permeation of steam throughout the waste.

The autoclave vessel is pressurized with saturated steam. The steam from the autoclave vessel is vented through a condenser at the end of the processing cycle.

A vacuum is again applied to the autoclave vessel following venting of the unit. The post vacuum cycle removes remaining moisture from the vessel and waste. This completes the processing cycle.

The inlet door is opened and the waste carts are removed from the autoclave following completion of the-processing cycle. The sterilized waste is allowed to cool and is then removed from the processing carts for disposal with the normal shop waste.

IV. Test Description

The Autoclave evaluation test equipment was set up on March 3, 2008. This included the Autoclave, Minicam, materials check, procurement of required test materials, thermocouple setup, steam line connection, and a pretest meeting.

The test runs started the morning of March 4, 2008.

Test Run 1 - DPE Suits, Whole

Feed Set-Up

The autoclave bin was set inside the autoclave and a high temperature plastic bin liner placed in the bin. The 6 mil bags had not arrived from TOCDF (The 6 mil polyethylene bags were coming via

FedEx from TOCDF). A standard 30 gallon Polyethylene garbage bag and a heavy duty 20 gallon zip lock bag were used to contain the DPE suits. Two bags were used instead of one as stated in the test procedure because of the small size of the bags. The bags were loaded with 2 ½ DPE suits each and two suits were placed around the bags in the bin for a total of 7 suits, approximately 75 lbs. The sachets were placed randomly in the bags to cover the bottom, middle and top areas. 1400 mg of surrogate material was then poured in the bottom, middle and top portions of the bags and on the loose suits in the bin.

Four thermocouples were randomly place around the bags for temperature monitoring.

Test Description

The autoclave temperature was set at 235°F. The autoclave steam cycle was set at 60 minutes. The autoclave steam jet air ejector was designed for 125 psig steam. The vacuum cycle set point was increased from -27"hg to approximately -18"hg because the shop steam supply only provided approximately 80 psig steam pressure.

The temperature and pressure were recorded manually during this test because the autoclave Temperature recorder was not functioning.

The bin was loaded at 0816 on 3/4/08 and the door closed.

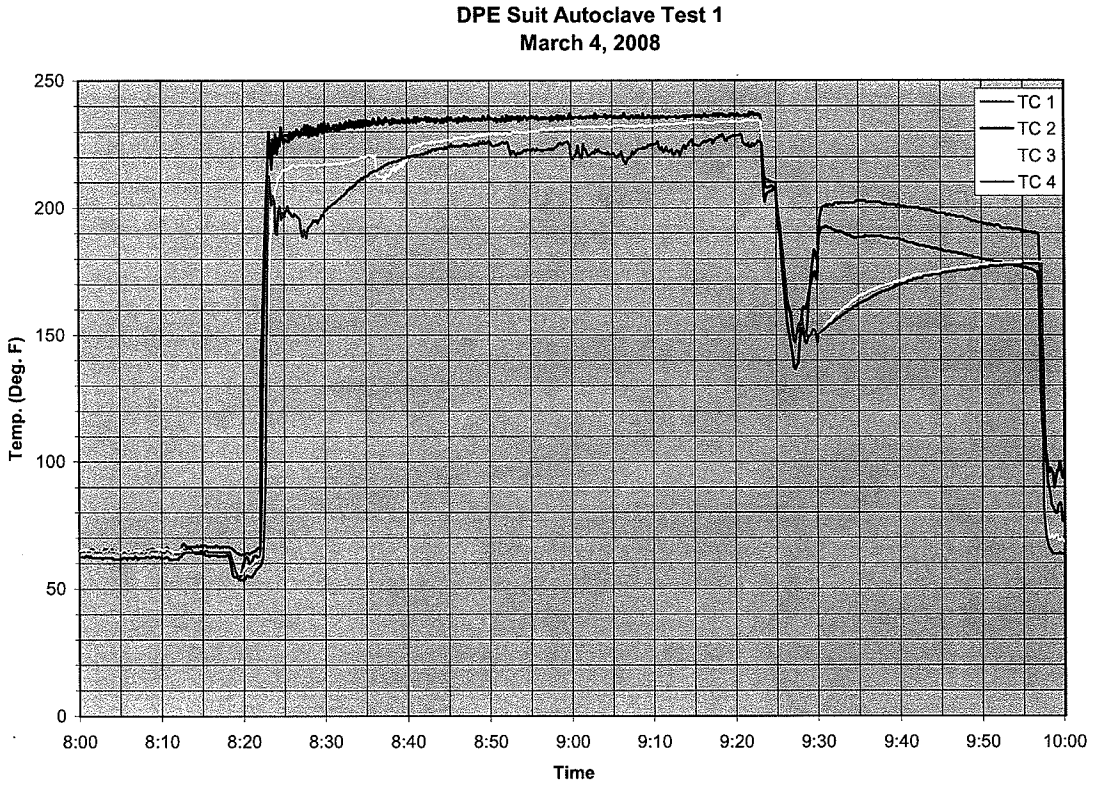
The autoclave cycle was started at 0818 on 3/4/08. The initial autoclave temperature at the start of the test was 63°F. The vacuum achieved during the pre-vacuum was -9 Psi or -18.3"hg.

The steam heat cycle was started at 0822.

Autoclave conditions recorded during the processing cycle:

Time	Temperature, °F	Pressure, Psig
0825	231	7.6
0835	235	9.0
0845	235	8.5
0855	235	8.6
0905	235	8.0
0915	235	8.2
0920	235	8.1

The following chart indicates the temperatures of the test thermocouples distributed around the suits.



The autoclave steam heat cycle was completed at 0922 on 3/4/08

The vent and the post vacuum cycle were completed at 0945 on 3/4/08 and the 15 minute hold time for the mini-cams test was started.

The mini-cams indicated surrogate present as vapor in the autoclave head space.

Date	≈Time		MINICAMS Result		1 VSL		Concentration
3/4/2008	0954 hrs	Test 1 Sample Concentration	= 1.95 VSL	X	$\frac{0.010417 \text{ mg/M}^3}{1 \text{ VSL}}$	=	0.020313 mg/M^3

The autoclave was open to inspect the load.

No deterioration or discoloration of the DPE suits was noted. The Polyethylene garbage bag and the heavy duty ziplok bags were intact and did not allow the steam from the autoclave to contact the DPE suits. The test was stopped and sachets collected based on these observations. The cycle time was not extended as the test temperature was not enough to allow the steam to contact the suits for decontamination. It was decided to increase the autoclave temperature to 250°F for the next DPE suit test due to the excellent condition of the DPE suits.

Extraction analysis

The sachets recovered from this test will not be subjected to laboratory analysis as they have not been exposed to steam during the processing cycle.

Test Run 2 - DPE Suits, Whole

Feed Set-Up

The autoclave bin was set inside the autoclave and a high temperature plastic bin liner placed in the bin. The 6 mil bags had not arrived from TOCDF (The 6 mil polyethylene bags were coming via FedEx from TOCDF). A standard 30 gallon Polyethylene garbage bag and a heavy duty 20 gallon zip lock bag were used to contain the DPE suits. Two bags were used instead of one as stated in the test procedure because of the small size of the bags. The bags were loaded with 2 ½ DPE suits each and two suits were placed around the bags in the bin for a total of 7 suits, approximately 75 lbs. The sachets were placed randomly in the bags to cover the bottom, middle and top areas. 1400 mg of surrogate material was then poured in the bottom, middle and top portions of the bags and on the loose suits in the bin.

Four thermocouples were randomly place around the bags for temperature monitoring.

Test Description

The autoclave temperature was set at 250°F. The autoclave steam cycle was set at 60 minutes. The autoclave steam jet air ejector was designed for 125 psig steam. The vacuum cycle set point was increased from -27"hg to approximately -18"hg because the shop steam supply only provided approximately 80 psig steam pressure.

The temperature and pressure were recorded manually during this test because the autoclave Temperature recorder was not functioning.

The bin was loaded at 1020 on 3/4/08 and the door closed.

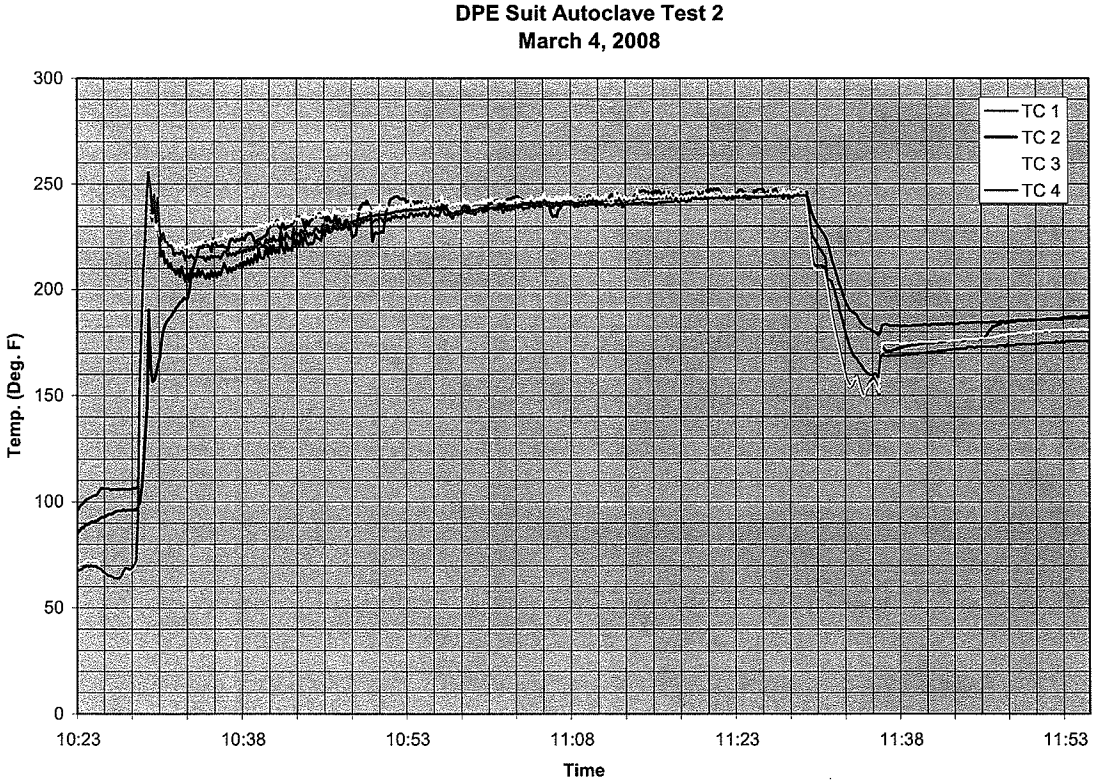
The autoclave cycle was started at 1024 on 3/4/08. The vacuum achieved during the pre-vacuum was -9 psi or -18.3"hg.

The steam heat cycle started at 1029.

Autoclave conditions recorded during the processing cycle:

Time	Temperature, °F	Pressure, Psig
1030	249	14.0
1040	250	14.8
1050	250	14.9
1060	250	14.9
1110	250	15.1
1120	250	14.6
1130	250	14.8

The following chart indicates the temperatures of the test thermocouples distributed around the suits.



The autoclave steam heat cycle completed at 1130 on 3/4/08

The vent and the post vacuum cycle was completed at 1137 on 3/4/08 and the 15 minute hold time for the mini-cams test was started.

The mini-cams indicated surrogate present as vapor in the autoclave head space.

Date	≈Time		MINICAMS Result		1 VSL		Concentration
3/4/2008	1159 hrs	Test 2 Sample Concentration	= 2.07 VSL	X	$\frac{0.010417 \text{ mg/M}^3}{1 \text{ VSL}}$	=	0.02156 mg/M^3

The autoclave was open to inspect the load.

No deterioration or discoloration of the DPE suits was noted. The Polyethylene garbage bag and the heavy duty ziplok bags were intact and did not allow the steam from the autoclave to contact the DPE suits. The test was stopped and sachets collected based on these observations. The cycle time was not extended as the test temperature was not high enough to allow the steam to contact the suits for decontamination. It was decided to cut the bags containing the DPE suits in order to allow the steam to contact the suits for the next test (Shredded DPE suits).

Extraction analysis

The sachets recovered from this test will not be subjected to laboratory analysis as they have not been exposed to steam during the processing cycle.

Test Run 3 - DPE Suits, Shredded

Feed set up

The autoclave bin was set inside the autoclave and a high temperature plastic bin liner placed in the bin. The 6 mil bags had not arrived from TOCDF (The 6 mil polyethylene bags were coming via FedEx from TOCDF). A standard 30 gallon Polyethylene garbage bag and a heavy duty 20 gallon zip lock bag were used to contain the DPE suits. Two bags were used instead of one as stated in the test procedure because of the small size of the bags. The bags were loaded with approximately 2 ½ DPE suits each (5 total), approximately 53 lbs. The DPE suit material varied in size, but the pieces were each approximately 6" X 6". The sachets were placed in the bags to cover the bottom, middle and top areas. 1400 mg of surrogate material was then poured in the bottom, middle and top portions of the bags. The bags were split to allow steam exposure to the suit material without the need to melt the bags.

Four thermocouples were randomly place between the pieces of suit material for temperature monitoring.

Test Description

The autoclave temperature was set at 250°F. The autoclave steam cycle was set at 60 minutes. The autoclave steam jet air ejector was designed for 125 psig steam. The vacuum cycle set point was increased from -27"hg to approximately -18"hg because the shop steam supply only provided approximately 80 psig steam pressure.

The temperature and pressure were recorded manually during this test because the autoclave Temperature recorder was not functioning.

The bin was loaded at 1234 on 3/4/08 and the door closed.

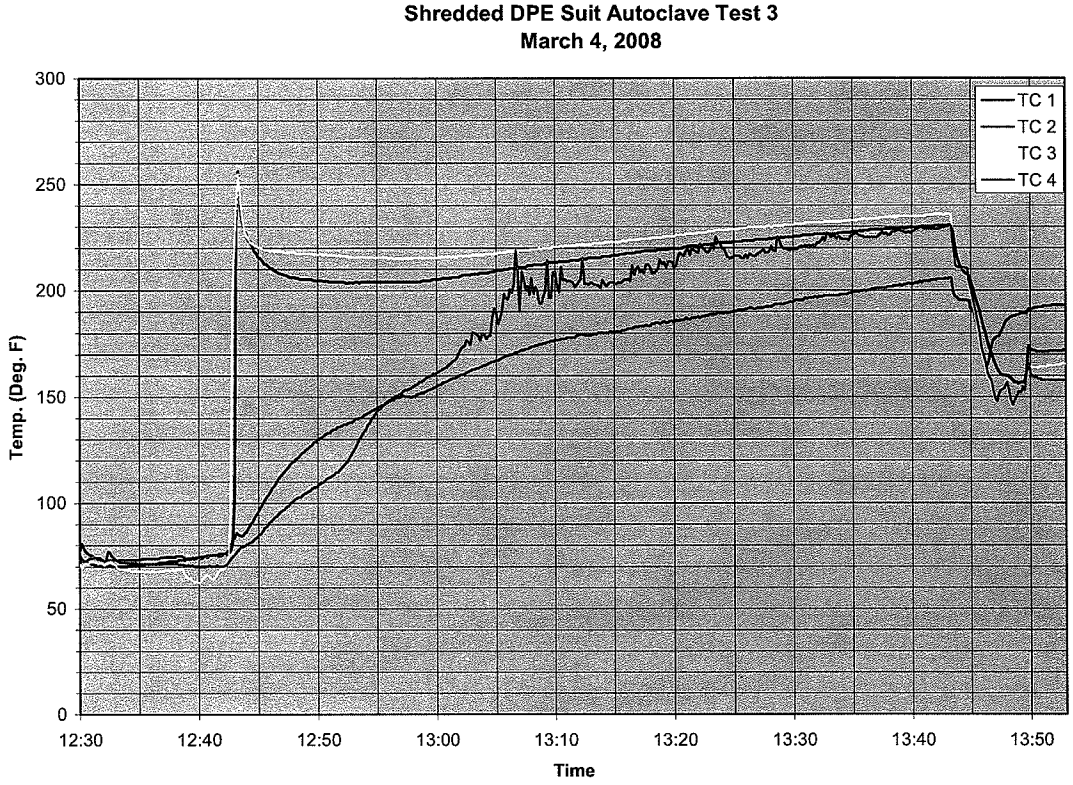
The autoclave cycle was started at 1238 on 3/4/08. The vacuum achieved during the pre-vacuum was -9 psi or -18.3"hg.

The steam heat cycle was started at 1242.

Autoclave conditions recorded during the processing cycle:

Time	Temperature, °F	Pressure, Psig
1243	235	12.7
1250	249	13.6
1300	250	14.3
1310	250	14.8
1320	250	14.9
1330	250	14.9
1340	250	14.9

The following chart indicates the temperatures of the test thermocouples distributed around the suits.



The autoclave steam heat cycle was completed at 1342 on 3/4/08

The vent and the post vacuum cycle were completed at 1350 on 3/4/08 and the 15 minute hold time for the mini-cams test was started.

The mini-cams indicated surrogate present as vapor in the autoclave head space.

Date	≈Time		MINICAMS Result		1 VSL		Concentration
3/4/2008	1424 hrs	Test 3 Sample Concentration	= 2.32 VSL	X	$\frac{0.010417 \text{ mg/M}^3}{1 \text{ VSL}}$	=	0.02417 mg/M^3

The autoclave was open to inspect the load.

No deterioration or discoloration of the DPE suits was noted. The Polyethylene garbage bag and the heavy duty ziplok bag were intact and open (Cut at start of test), which allowed the steam from the autoclave to contact the DPE suits. The test cycle was not continued. Additional DPE suit runs will be completed at the end of the scheduled test runs after further evaluation of the test time and temperature based on minicams results.

The test sequence was changed to run wood next, in place of the tap gear, as it was anticipated that a similar problem will exist with TAP gear. It was also decided to operate the minicams throughout the steam cycle in close proximity to the steam trap vent. This will allow detection of the surrogate material while processing.

Extraction analysis

The sachets recovered from this test were not subjected to laboratory analysis, based on results from additional testing completed after the scheduled test runs.

Test Run 4 (Test 5 in Test Plan) - Wood

Feed set-up

The autoclave bin was set inside the autoclave and a high temperature plastic bin liner placed in the bin. Wood 2X4's were cut in random lengths (8 to 18 inches) and placed randomly in the in the lined bin.

Four thermocouples were placed between the pieces of wood. Some of the sachets with DPE suit material were inadvertently placed in this load. The 10 wood sachets were placed in the load.

Test Description

The autoclave temperature set point was 250°F. The initial autoclave steam cycle was set at 60 minutes. The cycle was extended due to low temperature readings of the thermocouples and detection of surrogate by the minicams at the steam trap vent.

The bin was loaded at 1438 on 3/4/08 and the door closed.

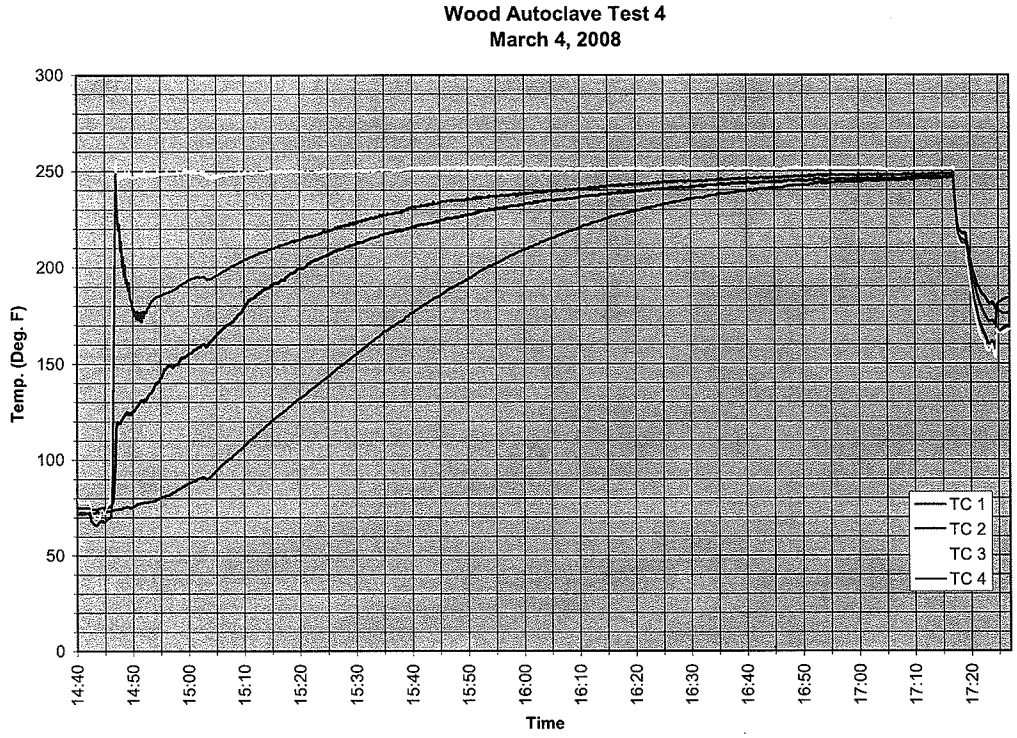
The autoclave operation was started at 1442 on 3/4/08. The vacuum achieved during the pre-vacuum was -9 psi or -18.3"hg.

The steam heat cycle started at 1447.

Autoclave conditions recorded during the processing cycle:

Time	Temperature, °F	Pressure, Psig
1450	249	13.0
1505	250	13.9
1515	250	13.9
1525	250	14.3
1535	250	14.1
1545	250	14.3
1555	250	14.9
1610	250	14.2
1625	250	14.1
1635	250	14.0
1655	250	13.9
1705	250	14.1
1715	250	14.0

The following chart indicates the temperatures of the test thermocouples distributed around the suits.



The autoclave steam heat cycle was completed at 1718 on 3/4/08.

The vent and the post vacuum cycles were completed at 1725 on 3/4/08 and the 15 minute hold time for the mini-cams test was started.

The mini-cams indicated surrogate present as vapor in the autoclave head space.

Date	≈Time	MINICAMS Result		1 VSL		Concentration
3/4/2008	1749 hrs	Test 4 Sample Concentration	= 1.13 VSL	X $\frac{0.010417 \text{ mg/M}^3}{1 \text{ VSL}}$	=	0.01177 mg/M^3

The autoclave was open to inspect the load.

The wood appeared dry with very little moisture, no discoloration.

Extraction analysis

Sample I.D.	Chloromethylnaphthalene µg/sample	Chloromethylnaphthalene DRE, percent
Wood-H-1	0.013	99.9993
Wood-H-2	0.506	99.9747
Wood-H-3	0.182	99.9909
Wood-H-4	0.086	99.9957
Wood-H-5	0.058	99.9971
	Average DRE % for Wood:	**99.9916**

Test Run 5 (Test 6 in Test Plan) - Charcoal

Feed set up

This test used three trays as identified in the test plan. The trays are each 24" square with 2" separation between them. Each tray contained approximately 14 lbs of charcoal for a total of 42 lbs of charcoal per load. Sachets were placed in each tray at the middle and corners using a total of 10 sachets.

Test Description

The autoclave temperature set point was changed from the test plan to be 290°F. The autoclave steam cycle was set at 60 minutes. Four thermocouples were place in the trays, two in the top tray, and one in the center of the middle and bottom tray.

The bin was loaded at 0745 on 3/5/08 and the door closed.

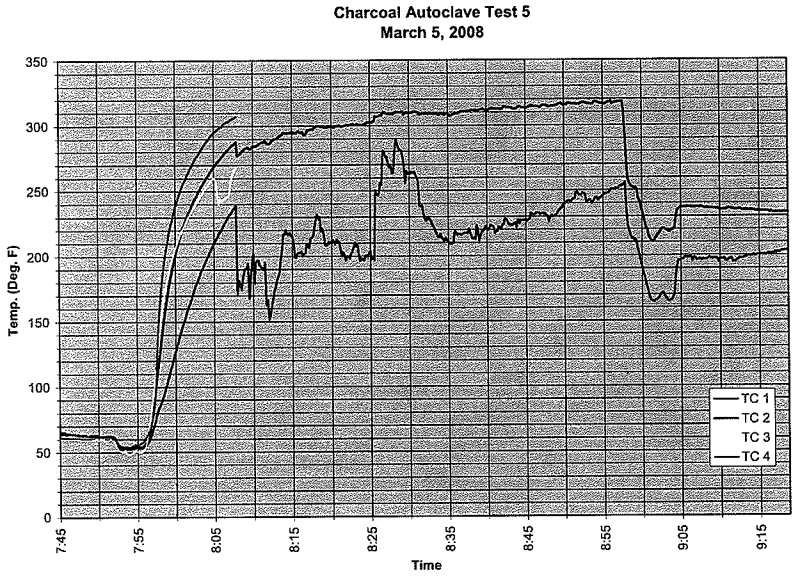
The autoclave operation was started at 0752 on 3/4/08. The vacuum achieved during the pre-vacuum was -9 Psi or -18.3"hg.

The steam heat cycle started at 0757.

Autoclave conditions recorded during the processing cycle:

Time	Temperature, °F	Pressure, Psig
0757	286	35.6
0805	289	41.1
0815	290	42.6
0830	291	42.7
0840	290	39.7
0850	290	39.1
0857	290	39.2

The following chart indicates the temperatures of the test thermocouples distributed in the trays.



The autoclave steam heat cycle completed at 0857 on 3/5/08.

The vent and the post vacuum cycle were completed at 0905 on 3/5/08 and the 15 minute hold time for the mini-cams test was started.

The mini-cams did not indicate a significant amount of surrogate present as vapor in the autoclave head space.

Date	≈Time	MINICAMS Result			1 VSL			Concentration	
3/5/2008	0930 hrs	Test 5 Sample Concentration	= 0.26	VSL X	0.010417	mg/M^3	=	0.00271	mg/M^3
					1	VSL			

The autoclave was open to inspect the load.

The top tray of charcoal had pock marks that may indicate boiling water out of the charcoal during the post vacuum cycle. The top tray did appear to be moist. The middle and the bottom trays appeared dry and had pock marks around the edges but not in the center. There was charcoal in the bottom of the autoclave that had been blown out of the tray during the process cycle.

Extraction analysis

Sample I.D.	Chloromethylnaphthalene µg/sample	Chloromethylnaphthalene DRE, percent
Carbon-H-1-1	0.0800	99.996
Carbon-H-1-2	0.0800	99.996
Carbon-H-1-3	0.0329	99.998
Carbon-H-1-4	0.0634	99.997
Carbon-H-1-5	0.0755	99.996
Average DRE % for Bulk Carbon Test 1:		**99.997**

Test Run 6 (Not Identified in test plan) - Blank DPE test

Feed set up

The 6 mil bags arrived from TOCDF. This test was conducted to determine if an autoclave processing temperature of 250°F will melt the bags that are used at TOCDF. Two clean uncontaminated DPE suits were place in a 6 mil polyethylene bag and closed with duct tape as is practice at TOCDF. No sachets were placed in the autoclave for this run.

Test Description

The autoclave temperature was set at 250°F. The autoclave steam cycle was set at 60 minutes. Three thermocouples were randomly placed around the bag and one was placed inside the bag.

The bin was loaded at 0948 on 3/5/08 and the door closed.

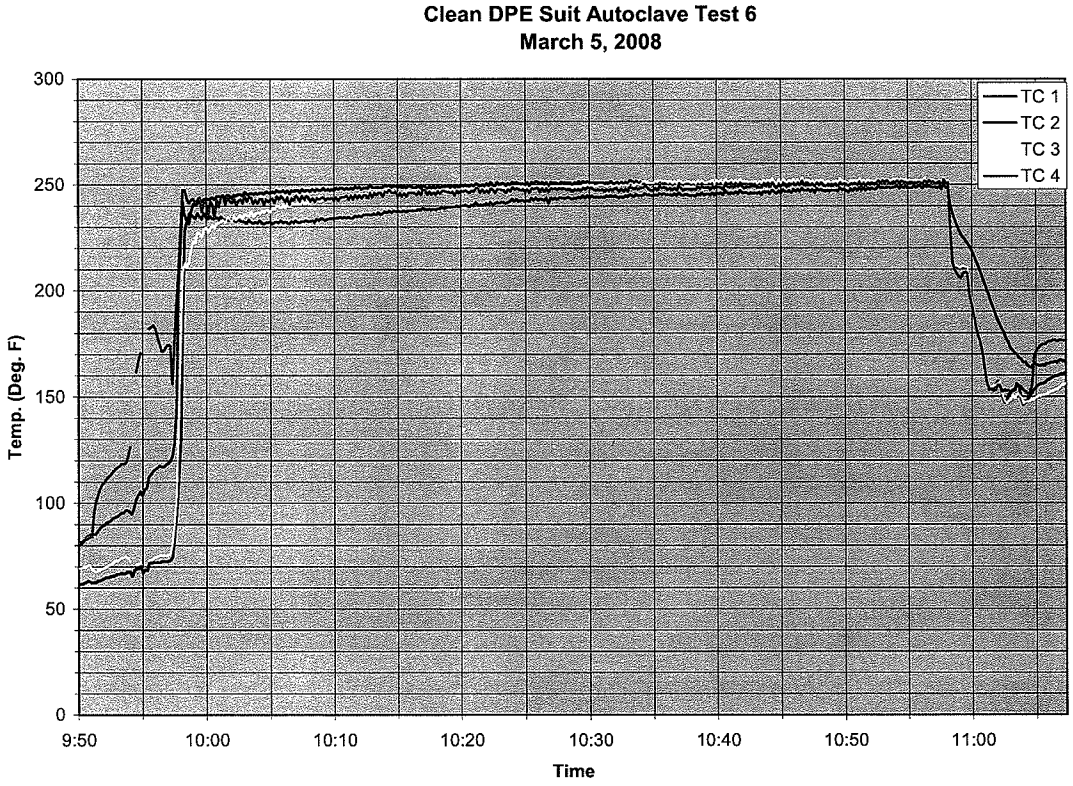
The autoclave operation was started at 0951 on 3/5/08. The vacuum achieved during the pre-vacuum was -9 Psi or -18.3"hg.

The steam heat cycle was started at 0959.

Autoclave conditions recorded during the processing cycle:

Time	Temperature, °F	Pressure, Psig
0959	246	12.2
1008	250	13.2
1015	249	13.4
1025	250	14.1
1035	250	14.2
1045	250	14.2
1055	250	14.1

The following chart indicates the temperatures of the test thermocouples.



The autoclave steam heat cycle was completed at 1058 on 3/5/08.

The vent and the post vacuum cycles were completed at 1106 on 3/5/08 and the 15 minute hold time for the mini-cams test was started.

The autoclave was open to inspect the load.

The bag had significantly degraded but only a very small hole could be found that may allow steam to penetrate the bag. There was some moisture in the bag, indicating that some steam penetration had occurred.

Test Run 7 - Charcoal

Feed set up

This test used three trays as identified in the test plan. The trays are each 24" square with 2" separation between them. Each tray contained approximately 14 lbs of charcoal for a total of 42 lbs of charcoal per load. Sachets were placed in each tray at the middle and corners using a total of 10 sachets.

Test Description

The autoclave temperature set point was changed from the test plan to 290°F. The autoclave steam cycle was set at 60 minutes. Four thermocouples were place in the trays, two in the top tray and one in the center of the middle and bottom tray.

The bin was loaded at 1305 on 3/5/08 and the door closed.

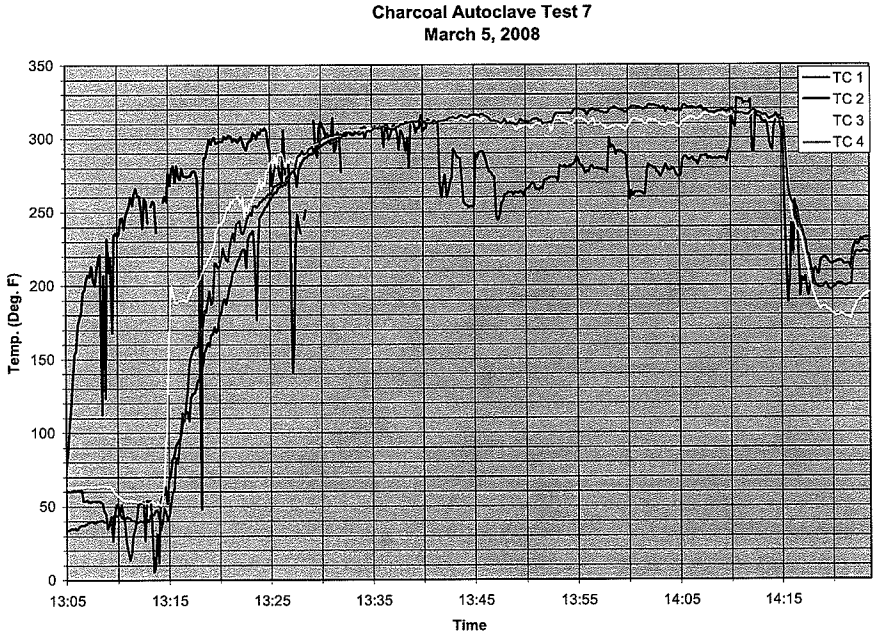
The autoclave operation was started at 1309 on 3/5/08. The vacuum achieved during the pre-vacuum was -9 psi or -18.3"hg.

The steam heat cycle was started at 1315.

Autoclave conditions recorded during the processing cycle:

Time	Temperature, °F	Pressure, Psig
1315	290	38.9
1330	290	39.4
1340	290	37.7
1350	290	37.5
1405	290	37.2

The following chart indicates the temperatures of the test thermocouples distributed on the trays,



The autoclave steam heat cycle was completed at 1415 on 3/5/08.

The vent and the post vacuum cycle was completed at 1422 on 3/5/08 and the 15 minute hold time for the mini-cams test was started.

The mini-cams did not indicate a significant amount of surrogate present as vapor in the autoclave head space.

Date	≈Time		MINICAMS Result		1 VSL		Concentration	
3/5/2008	1445 hrs	Test 6 Sample Concentration	= 0.33	VSL X	0.010417 / 1	mg/M^3 / VSL =	0.00344	mg/M^3

The autoclave was open to inspect the load.

The results were similar to those obtained in test run 6. The top tray of charcoal had pock marks that may indicate water boiling out of the charcoal during the post vacuum cycle. The top tray did appear to be moist. The middle and the bottom trays appeared dry and had pock marks around the edges, but not in the middle. There was charcoal in the bottom of the autoclave that had been blown out of the trays during the cycle.

Extraction analysis

Sample I.D.	Chloromethylnaphthalene µg/sample	Chloromethylnaphthalene DRE, percent
Carbon-H-2-1	0.0389	99.998
Carbon-H-2-2	0.0182	99.999
Carbon-H-2-3	0.0508	99.997
Carbon-H-2-4	0.0276	99.999
Carbon-H-2-5	0.0515	99.997
Average DRE % for Bulk Carbon Test 2:		**99.998**

Test Run 8 - Charcoal Filter

Feed set up

This test used one processing tray as identified in the test plan. The charcoal filter was placed on top of the processing tray. This allowed an air space around the entire filter. The filter was filled with charcoal directly in front of the autoclave. The sachets were placed at varying levels as the charcoal was added. Wires were placed on the sachets to allow removal without empting the charcoal filter. The filter was loaded with 46 lbs. of charcoal.

Test Description

The autoclave temperature set point was changed from the test plan to 290°F. The autoclave steam cycle was set at 60 minutes. Four thermocouples were place in the filter.

The tray was loaded at 1505 on 3/5/08 and the door closed.

The autoclave operation was started at 1510 on 3/5/08. The vacuum achieved during the pre-vacuum was -9 Psi or -18.3"hg.

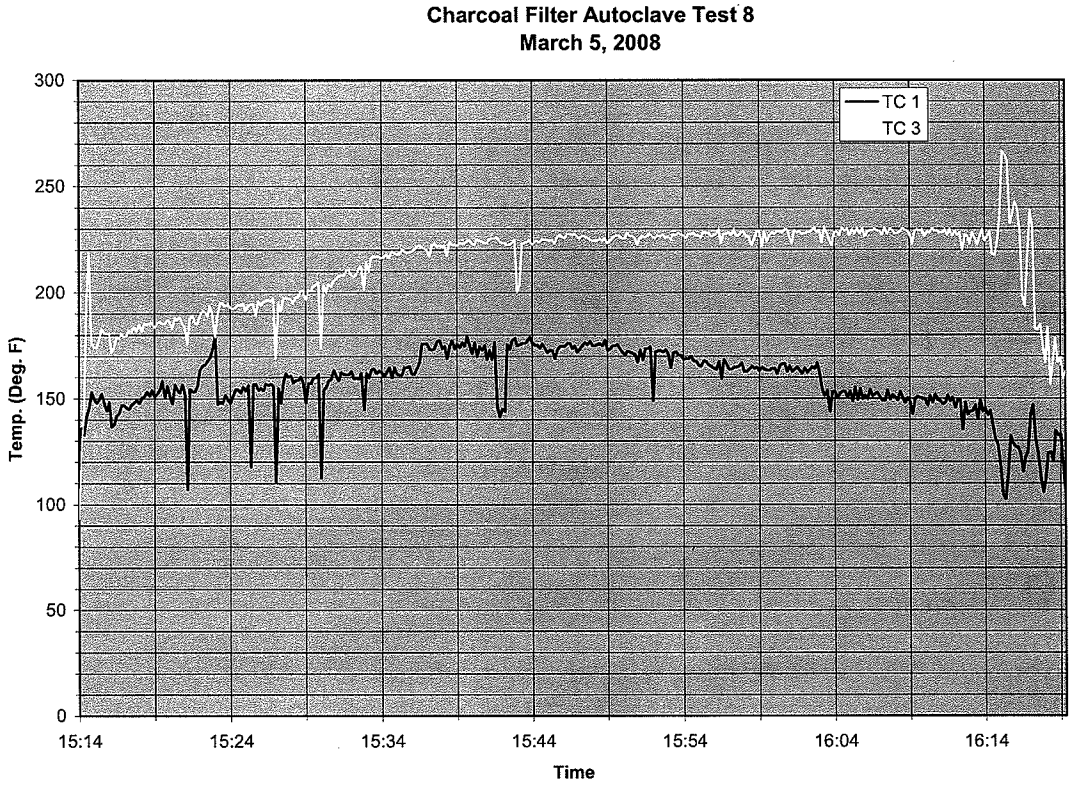
The steam heat cycle was started at 1515.

Autoclave conditions recorded during the processing cycle:

Time	Temperature, °F	Pressure, Psig
1515	290	40.7
1530	290	40.6
1540	290	40.9
1555	290	40.5
1609	290	40.4

The following chart indicates the temperatures of the test thermocouples distributed in the filter:

Note: TE's appear to be grounded through the autoclave on this test and do not indicate properly.



Note: TE's appear to be grounded through the autoclave on this test and do not indicate properly.

The autoclave steam heat cycle completed at 1615 on 3/5/08.

The vent and the post vacuum cycles were completed at 1642 on 3/5/08 and the 15 minute hold time for the mini-cams test was started.

The mini-cams did not indicate a significant amount of surrogate present as vapor in the autoclave head space.

Date	≈Time		MINICAMS Result		1 VSL		Concentration
3/5/2008	1645 hrs	Test 7 Sample Concentration	= 0.23	VSL X	$\frac{0.010417 \text{ mg/M}^3}{1 \text{ VSL}}$		= 0.002396 mg/M^3

The autoclave was open to inspect the load.

The charcoal filter cover was removed. The charcoal appeared dry and the sachets were removed without dumping out the charcoal.

Extraction analysis

Sample I.D.	Chloromethylnaphthalene µg/sample	Chloromethylnaphthalene DRE, percent
Carbon-H-3-1	0.0373	99.998
Carbon-H-3-2	0.0098	99.9995
Carbon-H-3-3	0.0253	99.999
Carbon-H-3-4	0.0508	99.997
Carbon-H-3-5	0.0298	99.999
Average DRE % for Carbon (Filter):		**99.998**

Test Run 9 (4 in Test Plan) - Tap gear

Feed set up

One 6 mil. Polyethylene bag was utilized to contain the tap gear. The bag was cut to allow steam penetration in case it did not deteriorate. The bag contained approximately 60 lbs of TAP gear (boots only).

Test Description

The autoclave temperature set point was changed from the test plan to be 290°F. The autoclave steam cycle was set at 60 minutes. Four thermocouples were placed at various points within the bagged material.

The bin was loaded at 0734 on 3/6/08 and the door closed.

The autoclave operation was started at 0735 on 3/6/08. The vacuum achieved during the pre-vacuum was -9 Psi or -18.3"hg.

The pre-steam heat cycle started at 0740. A steam leak was noticed around the autoclave door and the process stopped to correct the problem. The o-ring around the door had come out of its groove and was pinched between the door and the autoclave vessel. The o-ring was placed back in the groove and the seal area cleaned.

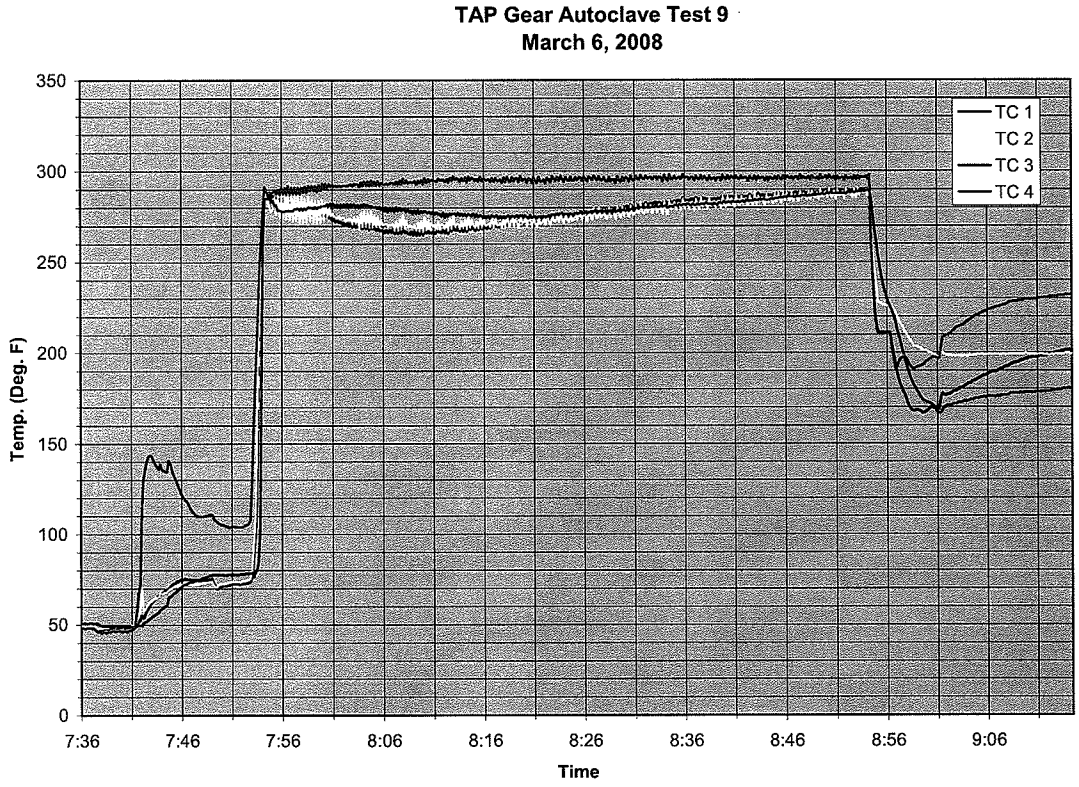
The autoclave operation was restarted at 0749 on 3/6/08.

The autoclave heat cycle was started at 0754 on 3/6/08.

Autoclave conditions recorded during the processing cycle:

Time	Temperature, °F	Pressure, Psig
0800	288	36.3
0812	291	40.1
0827	290	41.9
0845	290	41.0
0855	290	41.0

The following chart indicates the temperatures of the test thermocouples distributed around the boots:



The autoclave steam heat cycle was completed at 0901 on 3/6/08.

The vent and the post vacuum cycles were completed at 0910 on 3/5/08 and the 15 minute hold time for the mini-cams test was started.

The mini-cams did not indicate a significant amount of surrogate present as vapor in the autoclave head space.

Date	≈Time	MINICAMS Result		1 VSL		Concentration
3/6/2008	0930 hrs	Test 8 Sample Concentration	= 2.95 VSL	X $\frac{0.010417 \text{ mg/M}^3}{1 \text{ VSL}}$	=	0.030729 mg/M^3

The autoclave was open to inspect the load.

The polyethylene bag was significantly melted. The TAP boots were still in excellent condition.

Extraction analysis

Sample I.D.	Chloromethylnaphthalene µg/sample	Chloromethylnaphthalene DRE, percent
TAP-H-1	2.96	99.85
TAP-H-2	2.19	99.89
TAP-H-3	1.65	99.92
TAP-H-4	5.72	99.71
TAP-H-5	8.17	99.59
	Average DRE % for TAP Gear:	**99.79**

Test Run 10 (9 in Test Plan) - Dirt

Feed set up

This test used three trays as identified in the test plan. The trays are each 24" square with 2" separation between them. Each tray contained approximately 28 lbs of Dirt (Home Depot potting soil) for a total of 76 lbs of dirt per load. Sachets were placed in each tray at the middle and corners using a total of 10 sachets.

Test Description

The autoclave temperature set point was changed from the test plan to be 290°F. The autoclave steam cycle was set at 60 minutes. Four thermocouples were place in the trays, two in the top tray, and one in the center of the middle and bottom trays.

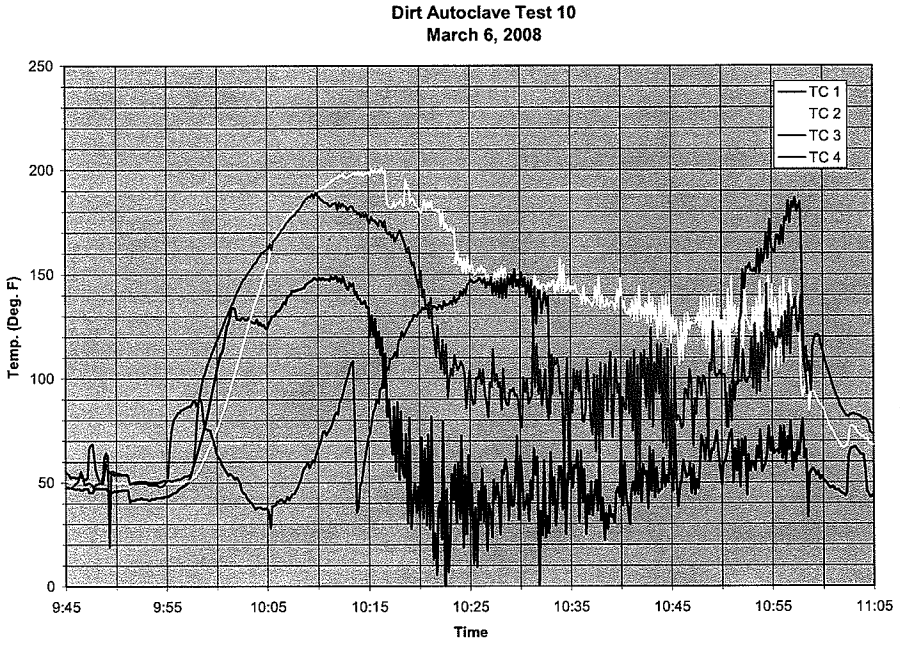
The autoclave operation was started at 0952 on 3/6/08. The vacuum achieved during the pre-vacuum was -9 Psi or -18.3"hg.

The autoclave heat cycle was started at 0957 on 3/6/08.

Autoclave conditions recorded during the processing cycle:

Time	Temperature, °F	Pressure, Psig
1000	289	40.5
1010	290	41.9
1020	290	41.6
1030	290	41.7
1045	290	41.3

The following chart indicates the temperatures of the test thermocouples distributed around the soil:
Note: TE's appear to be grounded through the autoclave on this test and do not indicate properly



The autoclave steam heat cycle was completed at 1057 on 3/6/08.

The vent and the post vacuum cycles were completed at 1104 on 3/6/08 and the 15 minute hold time for the mini-cams test was started.

The mini-cams did indicate a significant amount of surrogate present as vapor in the autoclave head space.

Date	≈Time		MINICAMS Result		1 VSL		Concentration
3/6/2008	1130 hrs	Test 9 Sample Concentration	= 1.71 VSL	X	0.010417 mg/M³ / 1 VSL	=	0.01781 mg/M³

The autoclave was open to inspect the load.

The dirt appeared moist and pock marks were noted in the upper tray and around the edges of the middle and lower trays. Indications were similar to those noted in the bulk charcoal tests.

Extraction analysis

Sample I.D.	Chloromethylnaphthalene µg/sample	Chloromethylnaphthalene DRE, percent
Soil-H-1	0.00308	99.99985
Soil-H-2	Not Detected (<0.001)	>99.99995
Soil-H-3	0.00328	99.99984
Soil-H-4	0.0046	99.99977
Soil-H-5	Not Detected (<0.001)	>99.99995
	Average DRE % for Soil:	**>99.99987**

Test Run 11 (duplicate of test 2 in Test Plan with some modifications) - DPE Suits

Feed set up

The autoclave bin was set inside the autoclave and a high temperature plastic bin liner placed in the bin. A polyethylene bag was loaded with approximately 10 lbs of DPE suit pieces. Sachets were placed in the bagged material and surrogate solution sprinkled as evenly as possible throughout the load. The polyethylene bag was cut to make sure the suits and the surrogate materials were exposed to the process steam. This test is intended to demonstrate if mass makes a difference in decontamination of the material.

Four thermocouples were randomly placed around the bag for temperature monitoring.

Test Description

The autoclave temperature set point was 250°F. The autoclave steam cycle was set at 60 minutes.

The bin was loaded at 1152 on 3/6/08 and the door closed.

The autoclave operation was started at 1155 on 3/6/08. The vacuum achieved during the pre-vacuum was -9 Psi or -18.3"hg.

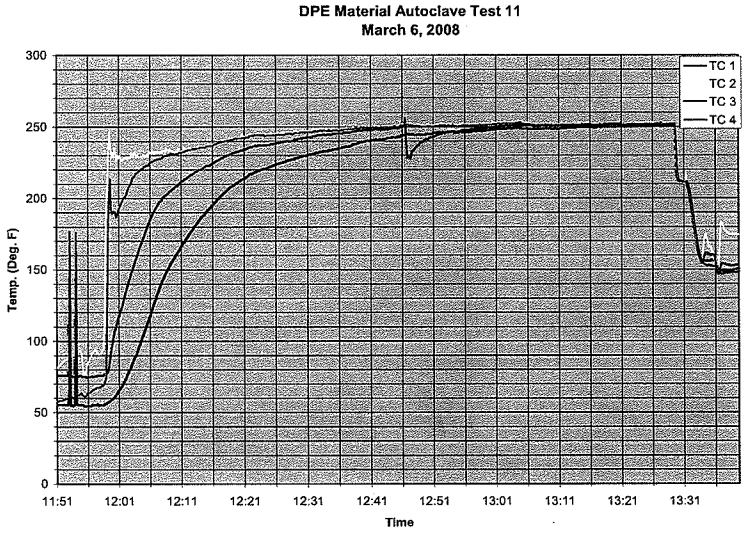
The steam heat cycle was started at 1200.

Autoclave conditions recorded during the processing cycle:

Time	Temperature, °F	Pressure, Psig
1210	249	15.0
1220	250	17.0
1230	250	14.7
1240	250	15.1
1250	250	14.6

At 1224 the TE's in the suits reached 220°F.

The following chart indicates the temperatures of the test thermocouples distributed in the suit material.



The autoclave steam heat cycle was completed at 1310 on 3/6/08.

The vent and the post vacuum cycles were completed at 1337 on 3/6/08 and the 15 minute hold time for the mini-cams test was started.

The mini-cams indicated surrogate present as vapor in the autoclave head space.

Date	≈Time		MINICAMS Result		1 VSL		Concentration
3/6/2008	1400 hrs	Test 10 Sample Concentration	= 2.73	VSL X	0.010417 mg/M³ / 1 VSL		= 0.028438 mg/M³

Preparations were made to restart cycle in an attempt to decontaminate suits.

The autoclave operation was re-started at 1402 on 3/6/08.

Started the autoclave heat soak at 1414 on 3/6/08.

Autoclave temperature was set at 250°F. Pressure was set at 14 to 15 psig.

Autoclave cycle was completed at 1515 on 3/6/08.

Final vent was completed at 1520 on 3/6/08.

The mini-cams indicated surrogate present as vapor in the autoclave head space.

Date	≈Time		MINICAMS Result		1 VSL		Concentration
3/6/2008	1545 hrs	Test 11 Sample Concentration	= 1.66	VSL X	0.010417 mg/M³ / 1 VSL		= 0.017292 mg/M³

Made preparations to restart cycle in an attempt to decontaminate suits.

Re-started the autoclave operation at 1547 on 3/6/08.

Started the autoclave heat soak at 1551 on 3/6/08.

Autoclave temperature was set at 250°F. Pressure was set at 14 to 15 PSIG

Autoclave cycle was completed at 1651 on 3/6/08.

Final vent was complete at 1658 on 3/6/08.

The mini-cams indicated surrogate present as vapor in the autoclave head space.

Date	≈Time		MINICAMS Result		1 VSL		Concentration
3/6/2008	1720 hrs	Test 12 Sample Concentration	= 1.32	VSL X	0.010417 mg/M³ / 1 VSL		= 0.01375 mg/M³

Completed testing for the day.

It is apparent that the surrogate vapor may not be hydrolyzing very quickly in the vapor phase based on the above testing. The autoclave chamber is vented and placed under a post vacuum but is not purged. Tomorrow morning we will try some quick tests to see if opening the autoclave door between cycles makes a difference.

Extraction analysis

Sample I.D.	Chloromethylnaphthalene µg/sample	Chloromethylnaphthalene DRE, percent
DPE-H-4-1	3.52	99.82
DPE-H-4-2	4.05	99.80
DPE-H-4-3	14.4	99.28
DPE-H-4-4	Not Detected (<0.6)	>99.97
DPE-H-4-5	4.50	99.78
Average DRE % for Shredded DPE:		**>99.73**

Test Run 12 (not in test plan) - Surrogate only in tray

Feed set up

This test used two trays as identified in the test plan. The trays are each 24" square with 2" separation between them. No waste medium was placed in the trays. Surrogate sachets and solution only were placed in a sample jar in the middle of the tray.

Test Description

The autoclave temperature set point was changed from the test plan to be 250°F. The autoclave steam cycle was set at 60 minutes. No thermocouples were used in this test.

The autoclave cycle was started at 0729 on 3/7/08. The vacuum achieved during the pre-vacuum was -9 Psi or -18.3"hg.

The autoclave heat cycle started at 0735 on 3/7/08.

Autoclave conditions recorded during the processing cycle:

Time	Temperature, °F	Pressure, Psig
0740	248	16.8
0800	250	16.0
0815	250	15.6
0830	250	15.2

The autoclave steam heat cycle was completed at 0835 on 3/6/08.

The vent and the post vacuum cycles were completed at 0841 on 3/6/08 and the 15 minute hold time for the mini-cams test was started.

The mini-cams indicated a significant amount of surrogate present as vapor in the autoclave head space.

Date	≈Time		MINICAMS Result		1 VSL		Concentration
3/7/2008	0905 hrs	Test 13-A Sample Concentration	= 2.54 VSL	X	$\frac{0.010417 \text{ mg/M}^3}{1 \text{ VSL}}$	=	0.026458 mg/M^3

The autoclave was open in an attempt to purge the vapor space. The door was opened for approximately 60 seconds and closed to allow another minicams sample. It was noted when the door was open that there was very little liquid left in the sample jar. The liquid may be condensate.

The following Minicams sample indicated a significant quantity of surrogate, although lower than the first measurement.

Date	≈Time		MINICAMS Result		1 VSL		Concentration
3/7/2008	0910 hrs	Test 13-B Sample Concentration	= 1.46 VSL	X	0.010417 mg/M³ / 1 VSL		= 0.015208 mg/M³

Extraction analysis

Sample I.D.	Chloromethylnaphthalene µg/sample	Chloromethylnaphthalene DRE, percent
DPE-H-5-1	60.3	96.98
DPE-H-5-2	23.8	98.81
DPE-H-5-3	118	94.09
DPE-H-5-4	130	93.49
DPE-H-5-5	112	94.39
Average DRE % for DPE Sachet:		**95.55**

Test Run 13 (not in test plan) - Uncontaminated DPE suit in Polyethylene bag

This test was conducted to determine if the DPE suits will hold up at a processing temperature of 270°F and if the 6 mil polyethylene bags will melt at this temperature.

Feed set up

Two uncontaminated DPE suits were loaded into one 6 mil polyethylene bag. The bag was tightly closed using duct tape similar to the closing method used at TOCDF. The autoclave bin was lined with the autoclave bin liner and the bag containing the DPE suits was place in the bin.

Test Description

The autoclave temperature set point was 270°F. The autoclave steam cycle was set at 60 minutes. No thermocouples were used in this test.

The autoclave cycle was started at 0932 on 3/7/08. The vacuum achieved during the pre-vacuum was -9 Psi or -18.3"hg.

The autoclave heat cycle was started at 0937 on 3/7/08.

Autoclave conditions recorded during the processing cycle:

Time	Temperature, °F	Pressure, Psig
1000	270	26.1
1010	270	26.4
1025	270	26.5

The autoclave steam heat cycle was completed at 1037 on 3/7/08.

The vent and the post vacuum cycles were completed at 1042 on 3/7/08 and the 15 minute hold time for the mini-cams test started.

The autoclave was open to inspect the load.

The bag had melted and the DPE suits were still intact.

Test Run 14 (duplicate of test 2 in Test Plan with some modifications) - DPE Suits

Feed set up

Two DPE suits were loaded into one 6 mil polyethylene bag. The bag was tightly closed using duct tape similar to the closing method used at TOCDF. The suits were contaminated with the surrogate naphthalene solution and the 10 sachets were placed throughout the bag. The autoclave bin was lined with an autoclave bin liner and the bag containing the DPE suits was place in the bin.

Test Description

The autoclave temperature set point was 280°F. The autoclave steam cycle was set at 60 minutes. No thermocouples were used in this test.

The autoclave cycle was started at 1106 on 3/7/08. The vacuum achieved during the pre-vacuum was -9 Psi or -18.3"hg.

The autoclave heat cycle was started at 1110 on 3/7/08.

The autoclave operated at 280°F and 32 to 33 Psig throughout the processing cycle.

The autoclave steam heat cycle was completed at 1210 on 3/7/08.

The vent and the post vacuum cycles were completed at 1217 on 3/7/08 and the 15 minute hold time for the mini-cams test was started.

The mini-cams indicated a significant amount of surrogate present as vapor in the autoclave head space.

Date	≈Time		MINICAMS Result		1 VSL		Concentration	
3/7/2008	1240 hrs	Test 14-A Sample Concentration	= 2.64	VSL X	0.010417 mg/M³ / 1 VSL	=	0.0275	mg/M³

The autoclave door was opened for one minute and closed for re-sampling.

Date	≈Time		MINICAMS Result		1 VSL		Concentration	
3/7/2008	1310 hrs	Test 14-B Sample Concentration	= 1.58	VSL X	0.010417 mg/M³ / 1 VSL	=	0.01646	mg/M³

The autoclave was open to inspect the load.

Inspection showed that the bag containing the DPE suits had melted, allowing the steam to access the suits. The DPE suits were still intact.

DPE suit following autoclave processing at 280°F.

Extraction analysis

Sample I.D.	Chloromethylnaphthalene µg/sample	Chloromethylnaphthalene DRE, percent
DPE-H-6-1	90.2	95.49
DPE-H-6-2	873	56.34
DPE-H-6-3	71.3	96.43
DPE-H-6-4	901	54.94
DPE-H-6-5	24.6	98.77
Average DRE % for Bulk DPE:		**80.39**

V. Sampling and Analysis

Samples from nine (9) tests conducted during the week of 3-7 March 2008 were analyzed by GC/MS/SIM for the agent surrogate chloromethylnaphthalene. Two sets of five sachets containing a small mass (1 to 4 grams) of the waste matrix were distributed among the bulk waste articles being autoclaved in each test. One set of five sachets was spiked at a "High" loading value of 2 milligrams of chloromethylnaphthalene per sachet. The second set of five sachets was spiked at a "Low" loading value of 1 microgram of chloromethylnaphthalene per sachet. A draft report summarizing the analytical results obtained by Southwest Research Institute® (SwRI®) from the samples collected during the series of autoclave tests is contained in appendix A.

A valid Destruction and Removal Efficiency (DRE) was not able to be calculated from the Low spike samples. The residual mass found on the majority of the sachets was either below the instrument detection limit or at a level comparable to the field blanks. The bulk waste in each test was also spiked with 1.4 grams of chloromethylnaphthalene mixed into 25 milliliters of toluene. The spiked toluene solution was sprinkled throughout the bulk waste items. The presence of this large quantity of chloromethylnaphthalene inside the autoclave further confounded interpretation of the "Low" spike sachet sample results. Some sachets yielded a residual mass greater than their theoretical spike values.

Only the analytical results from the High spike samples are felt to be representative of the DRE achieved by the autoclave relative to the different waste matrices. The analytical data from the High spike samples summarizing the residual masses of chloromethylnaphthalene recovered from the samples and the subsequent DRE values are presented for each test.

VI. Conclusions and Recommendations

The autoclave surrogate testing demonstrates the ability to decontaminate chemical weapons demilitarization secondary wastes in an autoclave. Based on these tests, there is a relatively wide range of decontamination levels that may be achieved, depending on the waste being processed. This may be due to the surrogate and / or carrier fluid selection. In order to eliminate results biased by the surrogate and / or carrier fluid selection, it is recommended that testing be conducted in an chemical agent certified laboratory using the chemical agents GB, VX and HD to determine the exact reaction of these chemicals. The surrogate chloromethylnaphthalene should also be tested without a carrier fluid under the same conditions as the chemical agents to identify the reaction differences between the surrogate and the chemical agents.

APPENDIX C

Section 2.0

J. Scott, R. Martinez,
Autoclave Secondary Waste VX on DPE and Wood,
Interim Report,
Southwest Research Institute,
San Antonio, TX,
September 30, 2008.

AUTOCLAVE SECONDARY WASTE
VX ON DPE AND WOOD

Interim Report

Prime Contract DACA87-89-C-0076
Subcontract G51972
SwRI® Project 01.14121

Prepared by:

Jim Scott
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October 3, 2008

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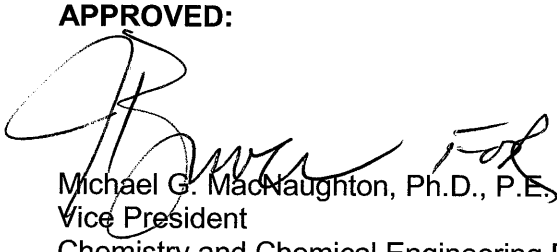
APPROVED:



Michael G. MacNaughton, Ph.D., P.E.
Vice President
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1. INTRODUCTION

The Tooele Chemical Agent Disposal Facility (TOCDF) is currently planning for processing of secondary and closure wastes. Installation of a system specifically for processing of secondary wastes would allow parallel processing of secondary wastes, increase the availability of the Metal Parts Furnace (MPF) to process munitions, and shorten the facility's overall schedule. In an earlier study, Continental Research & Engineering (CR&E) conducted an investigation of processing alternatives and found that the treatment of secondary waste through an autoclave is an attractive processing alternative due to its relative low cost and short implementation schedule. In addition, autoclaves have been used successfully by the Army for decontamination of BDO suits contaminated with chemical agents GB and VX, and Battelle has evaluated HD destruction in an autoclave environment for the Pueblo Chemical Agent-Destruction Pilot Plant Project (PCAPP).

In this project, Southwest Research Institute® (SwRI®) was tasked to conduct decontamination experiments using a commercial-off-the-shelf (COTS) bench model autoclave. The objectives of the test program were to: (1) obtain analytical results documenting the capability of an autoclave system to decontaminate secondary wastes contaminated with chemical agents (i.e., 'proof-of-concept' tests); and (2) document the operating/processing parameters (temperature, time, pressure, and vacuum) needed by the bench model autoclave to achieve a given level of decontamination performance.

To date, SwRI® has completed autoclave tests to treat DPE and wood contaminated with neat VX agent. Those results are summarized in this interim report. Testing of charcoal contaminated with VX, and all three waste matrices contaminated with chemical agents GB and HD, is continuing and will be reported in a final report.

2. EXPERIMENTAL APPROACH

2.1 Test Procedure

The experimental approach for the test program is detailed in "Test Plan for Barnstead – Harvey Autoclave Agent Testing," Draft – E, Revision 2, dated July 7, 2008. The Test Plan presents a detailed description of the operational and test procedures employed during the experiments, and it is referenced here for additional information.

A couple of modifications to the original test plan had to be adopted as the capabilities of the autoclave became apparent and impediments to the original vapor monitoring technique were discovered during early tests. These are discussed in Section 3.1. The modifications were necessary to yield reproducible test results indicative of the autoclave performance. A synopsis of the current testing procedure follows:

- A 10-gram sample of the secondary waste matrix (DPE, wood, or charcoal) is spiked with 900 micrograms (µg) of neat chemical agent.

- The spiked sample is placed on a tray inside the autoclave, positioned so that the sample is near the rear of the chamber, and the door is closed.

- After approximately 45 minutes, the agent vapor concentration inside the headspace of the autoclave is monitored using a near-real time monitor (MINICAMS).

- Since the autoclave is limited to a maximum exposure time of 99 minutes for a single cycle, the autoclave is operated at 275 °F for two, 90-minute exposure cycles using the "Unwrapped" program cycle.

- A 10 to 12 minute period of time elapses between the two exposure cycles to permit recovery of the water and vapor condensate discharges collected from the first cycle.

- At the conclusion of the second 90-minute exposure cycle, the agent vapor concentration inside the headspace of the autoclave is monitored using a near-real time monitor (MINICAMS).

- The spiked sample is recovered from the autoclave and assayed for residual agent content along with the liquid and vapor condensate discharges from both exposure cycles utilizing the analytical protocols referenced in the Test Plan.

- The CycleStor data files detailing the operating conditions for each of the two cycles are downloaded for documentation.

- Following a test, with the autoclave empty, the autoclave is operated for a 90-minute exposure cycle at 275 °F followed by a 30-minute drying cycle to purge any residual VX from the autoclave chamber headspace and the discharge lines prior to the next test. This purge cycle typically occurs at the end of the day and the autoclave door remains latched in the closed position, but unsealed, overnight. Headspace monitoring of the autoclave chamber prior to a test yields VX concentrations at baseline levels (typically 0.02 to 0.06 VSL).

2.2 Waste Discharges

Again, the Test Plan presents a detailed description of the procedures employed to collect the waste discharges from the autoclave for subsequent agent analyses. To reiterate, there are two waste discharges from the autoclave:

- Water – In the "Unwrapped" cycle, 400 mLs of deionized water is automatically added into the autoclave chamber during the pre-vacuum stage. The loading inside the chamber is very small (10 grams of waste) compared to the 12-pound maximum loading limit for the "Unwrapped" cycle (according to the manufacturer's Operation Manual). Thus, only a small percentage of this water volume can be adsorbed into the waste matrix (this water is recovered from the waste matrix during the drying stage). Consequently, at the end of the exposure cycle, approximately 250 mLs of water remains inside the chamber. This excess water is expelled from the chamber by the pressure (~ 31 to 32 psig) present inside the chamber.

- Vapor Condensate – During the pre-vacuum and drying stages, the autoclave vacuum pump is used to attain a negative pressure (~ -10 to -13 psig) inside the chamber. As the vapor is pulled from the chamber it passes through a heat exchanger upstream of the pump. The discharge from the vacuum pump consists of condensate and vapors.

The original plumbing of the autoclave directed both of the waste discharges into a polymeric waste tank. Since the intent was to analyze each waste discharge separately, the plumbing was modified to accomplish this task. Figure 1 presents a schematic of the waste discharge collection system. The water discharge is routed to two, 500-mL traps in series immersed in ice. The vapor condensate discharge is routed to an identical 500-mL trap, followed by a 500-mL impinger filled with 150 mL of triacetin; again both vessels are immersed in ice. Please note that Figure 1 does not show the plumbing configuration that

enabled the MINICAMS to directly monitor the headspace of the autoclave chamber. As discussed in the next section, this approach for the headspace monitoring had to be abandoned, and the plumbing was removed at the end of the initial test series. Hence, Figure 1 illustrates the system, as it existed for the 'official' performance tests.

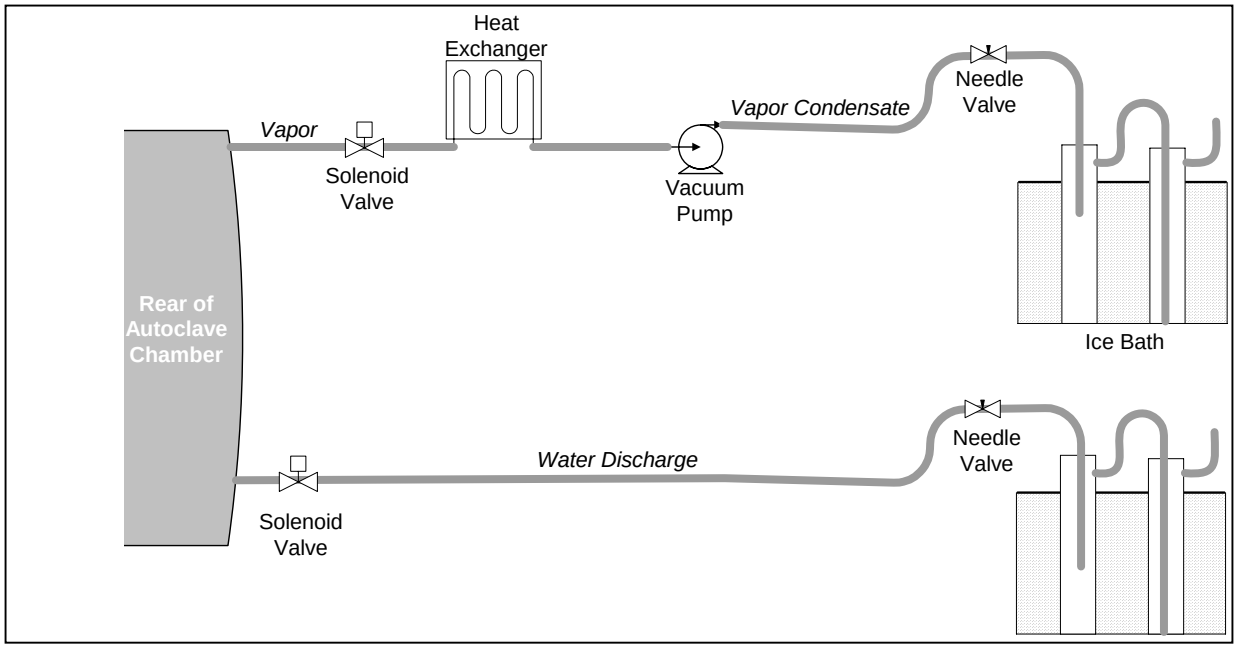

Figure 1. Waste discharge collection system.

3. FINDINGS

3.1 Initial VX Tests

A series of six (6) tests with VX agent were performed during which 'lessons learned' were discovered concerning the autoclave performance and the VX vapor monitoring technique. Subsequent modifications to the Test Plan were adopted to alleviate the discovered deficiencies. These initial tests are described below solely to document the rationale for the Test Plan modifications. The results for the remaining tests, as presented in the next section, represent the 'official' performance results obtained by the autoclave using the operating procedures as summarized above.

It is stressed that during the first five tests, the pre- and post-exposure vapor monitoring technique for the autoclave chamber headspace was implemented exactly as described in the Test Plan. That is, the chamber headspace was monitored directly by the MINICAMS using a port located in the rear of the chamber (see Figure 2). A series of two, 3-way valves were utilized to a vacuum pump to pull the chamber headspace sample into the MINICAMS during the pre- and post-exposure monitoring periods. During the autoclave cycle, the valves were switched to route the vapor discharge to the heat exchanger while the MINICAMS monitored the air inside the glove box.

For the pre-exposure monitoring, the autoclave door remained sealed (i.e., the chamber remained completely closed from the time the spiked sample was loaded into chamber until the monitoring began).

For the post-exposure monitoring, the automatic program sequence for the autoclave unseals the door at the conclusion of the drying period. However, the door remains latched in the closed position, and the MINCAMS pulled the headspace sample from the rear of the chamber.

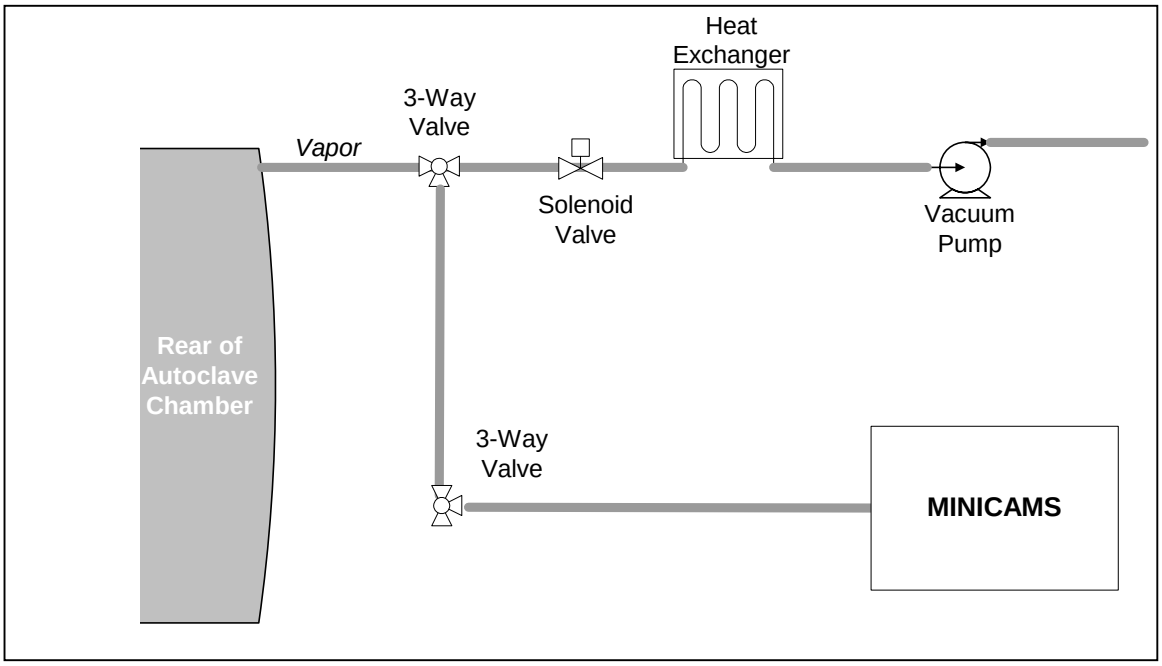

Figure 2. Direct headspace monitoring of autoclave chamber using MINICAMS.

3.1.1 VX – DPE Test #1

Preliminary tests performed by SwRI spiking onto wood with VX had indicated that a loading of approximately 400 µg of neat agent should yield a desirable vapor concentration within the 20-Liter chamber headspace (targeting a concentration of ~ 10 VSL). However, using this dosage mass in the first test with a 10-gram swatch of DPE (20-mil) only yielded a pre-exposure VX vapor concentration of 5.0 VSL. The autoclave cycle for this test was performed as detailed in the Test Plan: an "Unwrapped" cycle with a 60-minute exposure period at 275 °F followed by a 30-minute drying period. Following the conclusion of the drying period, the post-exposure VX vapor concentration inside the headspace was 0.48 VSL. Although the post-exposure vapor concentration was acceptable (i.e., < 1.0 VSL), the lower-than-anticipated vapor concentration obtained prior to initiation of the autoclave cycle caused us to seek a higher spiking level for the subsequent tests.

3.1.2 VX – DPE Tests #2 & #3

In these two tests, the autoclave operating conditions remained unchanged, but the spike level was increased to 900 µg of neat VX. Despite the increase, the pre-exposure VX headspace concentrations remained low (0.76 and 3.3 VSL for tests #2 and #3, respectively). The post-exposure vapor concentrations (0.32 and 0.57 VSL for tests #2 and #3, respectively) continued to show that the 60-minute exposure period and 30-minute drying period was yielding satisfactory results. The decision was made to keep the dosage at 900 µg for the upcoming tests of VX on wood.

3.1.3 VX – Wood Tests #1 & #2

Short segments of wooden dowels (untreated, 3/8-inch diameter) totaling 10 grams were used for the wood tests. A shallow depression was punched into the side of one of the dowel segments as a 'receptacle' for the VX spike. The spike loading remained at 900 µg and the autoclave operating conditions remained unchanged from the DPE tests. The pre-exposure VX vapor concentrations inside the chamber were comparable to the levels seen in the DPE tests (3.0 and 1.2 VSL for tests #1 and #2, respectively).

The post-exposure VX vapor concentration in Test #1 was 0.46 VSL, however, numerous interferences were observed in the MINICAMS chromatogram that may have affected measurement of the true agent concentration. Following the test, the heat-trace sampling line for the MINICAMS was cleaned and droplets of water were observed near the AgF conversion pad. The AgF conversion pad was replaced and additional heat tape was wrapped around the vapor sampling line at the rear of the autoclave chamber.

The post-exposure VX vapor concentration in Test #2 was 3.49 VSL, however, the validity of this measurement was uncertain, as the interferences were even worse than observed in the previous test despite the additional heat tape and line cleaning. Further investigation of the sampling line revealed that the AgF conversion was soaked by condensed water. Flushing the sampling line with solvent revealed traces of residue. It was apparent that moisture from the autoclave chamber was capable of entering the MINICAMS sample line, and that this moisture was gradually fouling the line and the AgF pad over successive cycles.

Therefore, to obtain reliable vapor concentration measurements, the monitoring approach given in the Test Plan (and illustrated in Figure 2) had to be abandoned. The pre- and post-exposure vapor monitoring of the chamber headspace is currently accomplished by inserting the MINICAMS sample line directly into the chamber via the door. A short length of ¼-inch stainless steel tubing is placed on the end of the MINICAMS heat-trace line, with an AgF conversion pad positioned on the distal end of the steel tubing. The length of the steel tubing is just long enough to place the AgF conversion pad in the middle of the autoclave chamber. To obtain the pre-and post-exposure vapor concentrations, the autoclave door is cracked open just wide enough to slip the ¼-inch stainless steel tubing past the door, and then the door is pressed tightly against the tubing.

3.1.4 VX – DPE Test #4

With the change to the vapor monitoring protocol, an additional test was performed using spiked DPE. The purpose of the test was to ascertain whether the changed protocol affected the promising results that were observed during the first three tests. This test did show that the vapor readings obtained by the MINICAMS utilizing the sampling line connected into the earlier tests were misleading:

- Spiking 900 µg of VX onto the DPE sample yielded a pre-exposure VX vapor concentration in the chamber headspace of 12.5 VSL, more than double the level seen in any of the prior measurements.

- After the completion of the 60-minute exposure period and 30-minute drying period, the post-exposure VX vapor concentration inside the chamber was 13.2 VSL. A second measurement collected 35 minutes later yielded a VX vapor concentration of 3.0 VSL.

- The autoclave cycle was repeated (60-minute exposure period and 30-minute drying period) on this same sample. The post-exposure VX vapor concentration inside the chamber following the second cycle was initially 3.3 VSL, with a second reading of 1.30 VSL obtained 14 minutes later.

The conclusions derived from these early tests were:

- The plumbing arrangement devised to enable the MINICAMS to directly sample the autoclave chamber obviously caused erroneously low VX vapor concentration measurements at both the pre- and the post-exposure time periods.

- Although cracking the door slightly ajar to insert the sample line into the autoclave chamber is less than ideal, it afforded more reliable vapor concentration measurements than the protocol originally specified in the Test Plan.

- The time required to achieve acceptable VX vapor concentrations (<1.0 VSL) inside the chamber was greater than could be achieved by a single cycle (limited by the autoclave program to 99 minutes). The decision was made to operate two consecutive cycles, each comprised of a 90-minute exposure period and a 30-minute drying period, for each test.

- The water and vapor condensate discharges for each of the two cycles in a test are collected at the completion of their respective cycle and analyzed separately (Tables 3 and 6).

3.2 Triplicate VX Test Results for DPE

Table 1 presents the headspace vapor measurements obtained for the 'official' triplicate autoclave tests performed with 900 µg of neat VX agent spiked onto 10-gram swatches of 20-mil DPE. To re-iterate, each test consists of two consecutive cycles, each comprised of a 90-minute exposure period and a 30-minute drying period. The post-exposure vapor concentration is obtained immediately after the completion of the drying period in the second cycle. In addition, a second post-exposure vapor concentration measurement is collected 28 minutes after the initial measurement (the MINICAMS sample/purge cycle for VX is 7 minutes; collecting a sample after 4 cycles was a somewhat arbitrary selection, but it approximated a 30-minute 'hold' period at the conclusion of a cycle that possibly might be imposed upon an operational autoclave before the door was opened).

Table 1. VX Vapor Headspace Concentrations inside Autoclave Chamber for DPE Spiked with 900 Micrograms of VX

Test	Pre-Exposure Concentration, VSL	Initial Post-Exposure Concentration, VSL	Post-Exposure + 28 Minutes Concentration, VSL
1	11.0	1.49	0.68
2	15.9	1.01	0.31
3	16.9	0.44	< 0.06

Table 2 summarizes: (1) the residual VX agent found in the spiked DPE sample after undergoing the two successive autoclave cycles in a test, and (2) the DRE percentage calculated by comparing the residual VX mass to the spike mass of 900 µg.

Table 2. VX DRE for DPE Spiked with 900 Micrograms of VX

Test	Residual VX in DPE, micrograms	DRE, percent
1	0.055	99.994
2	0.100	99.989
3	0.249	99.972

Table 3 summarizes: (1) the volumes of water and vapor condensate collected at the conclusion of the two individual autoclave cycles in each test, (2) the concentration of VX agent found in the water and vapor condensate samples, and (3) the total mass of VX agent in the water and vapor condensate samples.

Table 3. VX in Discharges for DPE Spiked with 900 Micrograms of VX

Test	Cycle	Discharge	Volume, mL	VX concentration, nanograms/mL	Mass of VX, micrograms
1	1	Water	265	6.94	1.84
		Condensate	160	22.3	3.57
	2	Water	290	2.43	0.71
		Condensate	155	3.44	0.53
2	1	Water	265	7.48	1.98
		Condensate	160	21.2	3.40
	2	Water	270	3.22	0.87
		Condensate	155	3.81	0.59
3	1	Water	260	8.25	2.15
		Condensate	155	26.0	4.03
	2	Water	280	3.69	1.03
		Condensate	160	5.91	0.94

A discussion of these results will follow the presentation of the VX and wood test results.

3.3 Triplicate VX Test Results for Wood

Table 4 presents the headspace vapor measurements obtained for the 'official' triplicate autoclave tests performed with 900 µg of neat VX agent spiked onto 10-grams of wooden dowel segments. To re-iterate, each test consists of two consecutive cycles, each comprised of a 90-minute exposure period and a 30-minute drying period. The post-exposure vapor concentration is obtained immediately after the

completion of the drying period in the second cycle. In addition, a second post-exposure vapor concentration measurement is collected 28 minutes after the initial measurement.

Table 4. VX Vapor Headspace Concentrations inside Autoclave Chamber for Wood Spiked with 900 Micrograms of VX

Test	Pre-Exposure Concentration, VSL	Initial Post-Exposure Concentration, VSL	Post-Exposure + 28 Minutes Concentration, VSL
1	39.3	0.63	0.16
2	37.0	0.73	< 0.06
3	46.6	0.55	0.07

Table 5 summarizes: (1) the residual VX agent found in the spiked DPE sample after undergoing the two successive autoclave cycles in a test, and (2) the DRE percentage calculated by comparing the residual VX mass to the spike mass of 900 µg.

Table 5. VX DRE for Wood Spiked with 900 Micrograms of VX

Test	Residual VX in Wood, micrograms	DRE, percent
1	6.42	99.29
2	1.21	99.87
3	4.58	99.49

Table 6 summarizes: (1) the volumes of water and vapor condensate collected at the conclusion of the two individual autoclave cycles in each test, (2) the concentration of VX agent found in the water and vapor condensate samples, and (3) the total mass of VX agent in the water and vapor condensate samples.

3.4 EA2192 Tests

During the DPE and wood tests, SwRI was tasked to evaluate whether the VX degradation product EA2192 was present either in the autoclaved waste matrix or in the waste discharges. To detect and quantify EA2192, SwRI utilized a high performance liquid chromatography/mass spectrometry (HPLC-MS) analytical procedure previously developed to quantify agent decomposition products (ADPs) for the JACADS closure. Since this procedure requires extraction of solid matrices using HPLC-grade water, the EA2192 analysis could not be accomplished on the same waste sample analyzed for residual VX (i.e., the latter requires extraction with IPA/DCM solvent). Thus, separate autoclave tests had to be performed for the EA2192 analyses.

Table 6. VX in Discharges for Wood Spiked with 900 Micrograms of VX

Test	Cycle	Discharge	Volume, mL	VX concentration, nanograms/mL	Mass of VX, micrograms
1	1	Water	270	6.05	1.63
		Condensate	157	12.5	1.97
	2	Water	280	5.26	1.47
		Condensate	160	2.93	0.47
2	1	Water	262	9.50	2.49
		Condensate	158	11.2	1.77
	2	Water	250	6.88	1.72
		Condensate	162	3.05	0.49
3	1	Water	260	8.15	2.12
		Condensate	155	16.5	2.56
	2	Water	280	6.68	1.87
		Condensate	160	3.97	0.64

Two tests were performed for the EA2192 evaluations – one with DPE and one with wood dowels. The autoclave operational parameters were identical to the prior VX tests (two consecutive cycles, each comprised of a 90-minute exposure period and a 30-minute drying period) and the VX spiking mass remained unchanged (900 micrograms). The EA2192 results are presented in Table 7. No detectable concentrations of EA2192 were measured in any of the spiked waste samples or in the discharges.

Table 7. EA2192 Results for DPE & Wood Spiked with 900 Micrograms of VX

Sample I.D.	Volume, mL	EA2192 ppb (ng/mL)	EA2192, micrograms
10-grams DPE spiked w/900 ug VX	-	< 2	< 0.10
DPE, Water Cycle 1	260	< 2	< 0.52
DPE, Water Cycle 2	259	< 2	< 0.52
DPE, Condensate Cycle 1	165	< 2	< 0.33
DPE, Condensate Cycle 2	160	< 2	< 0.32
10-grams wood spiked w/900 ug VX	-	< 2	< 0.10
Wood, Water Cycle 1	260	< 2	< 0.52
Wood, Water Cycle 2	275	< 2	< 0.55
Wood, Condensate Cycle 1	162	< 2	< 0.32
Wood, Condensate Cycle 2	159	< 2	< 0.32

4. DISCUSSION

Some general observations concerning the tests:

- The collection traps, immersed in ice, used to collect the water and vapor condensate discharges yielded remarkably comparable volumes among the 12-autoclave cycles that comprised the triplicate series of DPE and wood tests.

    - Two traps in series are used to collect the water discharge from the chamber that occurs at the conclusion of the exposure period. Despite being a single discharge event under pressure (~ 31 to 32 psig) and at temperature (275 ºF), all but 5 to 10 mLs of the water is collected in the first trap. The installation of a needle valve to limit the flow rate of the water discharge from the autoclave chamber is largely responsible for the efficient collection by the first trap.

    - In contrast to the water discharge, the vapor condensate discharge consists of a slow, trickle that occurs intermittently during the pre-vacuum and the drying stages. It was apparent that the heat exchanger on the autoclave efficiently condenses the water vapor to enable effective collection by the discharge trap.

- The operational data collected by the CycleStor data system showed, for the DPE and wood tests, that the autoclave operated in a fairly repeatable manner. Chamber temperatures during the exposure stages were typically in the range of 276 to 277 ºF at pressures of 31 to 32 psig. Subsequent to these tests, a lag thermometer was inserted into the chamber to confirm chamber temperatures. This calibrated instrument showed that the actual chamber temperature was nominally a couple of degrees higher than indicated by the CycleStor data.

Regarding the analytical results for the tests:

- The average post-exposure VX vapor headspace concentration immediately following the conclusion of the drying stage for the triplicate DPE tests was close to the 1.0 VSL target (0.98 VSL average). Within 28 minutes, the VX vapor concentration inside the chamber was comfortably below the 1.0 VSL criteria.

- In the wood tests, the post-exposure VX vapor headspace concentration immediately following the conclusion of the drying stage was below the 1.0 VSL limit in all 3 tests.

- The DRE for the DPE tests exceeded 99.9 percent, while the wood tests yielded a DRE of greater than 99.0 percent.

- The VX agent trends for the water and condensate discharges differed between the two test substrates.

    - In the DPE tests:

        - The cumulative mass of VX found in the two discharges during the first cycle was consistently greater (by a factor of at least 3) than the total mass collected from both discharges in the second cycle. This would indicate that

most of the VX emissions occurred during the first cycle, which is a reasonable expectation.

- Furthermore, in the first cycle of the DPE tests, the mass of VX in the condensate was approximately double the mass detected in the water discharge.

- In the second cycle of the DPE tests, the masses of VX in the condensate and water discharges were very similar to one another.

- In the wood tests, however:

  - While the mass of VX discharged during the first cycle was greater than the second cycle, the difference was not as significant as seen in the DPE tests. This would indicate that the VX release during the second cycle was still prominent and probably the impetus for a lower DRE compared to DPE.

  - There was little disparity between the masses of VX detected in the condensate compared to that observed in the water discharges during the first cycle of the wood tests.

  - Finally, while the mass of VX in the condensate dropped significantly during the second cycle of the woods tests, there was only a small decrease in the mass of VX found in the water discharged in the second cycle compared to the first cycle.

5. CONCLUSION

The initial tests yielded information regarding the length of the exposure cycles to achieve VX vapor headspace concentrations near the 1.0 VSL targeted criteria. These tests also showed that some modification to the originally envisioned headspace monitoring approach was needed to obtain reliable VX vapor concentrations.

Once the cycle lengths and headspace monitoring methodology were refined, the subsequent experiments yielded insight into the performance results associated with autoclaving wood and DPE contaminated with neat VX agent under the defined test conditions. The extrapolation of these results to a full-scale, operational system is beyond the scope of this 'proof-of-concept' experimental program.

One cautionary note is made regarding the interpretation of the residual VX mass present in the 10-gram waste samples following the two consecutive autoclave cycles (Tables 2 and 5). Using the 10-gram weight of the spiked sample to calculate a residual VX concentration (e.g., ppb of VX in the 'treated' waste) could be misconstrued or deceptive regarding the autoclave performance capability. The 900 micrograms of neat VX occupied a minute portion of the surface area represented by the 10-gram sample. If the waste samples had been even larger (i.e., up to the maximum load limit of 12 pounds for this autoclave model), the identical spike loading likely would have yielded about the same residual mass of VX after treatment, but it would equate to a much lower VX residual on a concentration (ppb) basis.